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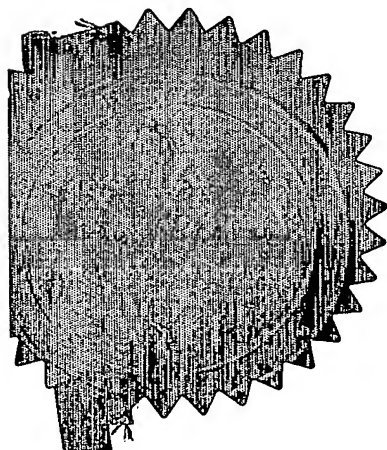
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Signed *Stephen Hordley*  
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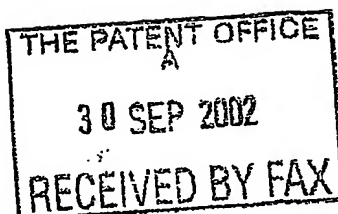
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BACCHUS TECHNOLOGIES LTD  
Incorporated in the United Kingdom  
The Cider Mill, Fair Oaks Farm  
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ADP No. 08572836002

PATENTS ACT 1977

PATENTS FORM No 1/77

The Comptroller,  
The Patent Office30SEP02 E7517B4-1 002626  
P01/7700 0.00-0222543.1

## REQUEST FOR GRANT OF A PATENT

0222543.1

30 SEP 2002

THE GRANT OF A PATENT IS REQUESTED BY THE UNDERSIGNED ON THE  
BASIS OF THE PRESENT APPLICATION

I Agent's reference: TAYLOR II

II Title of Invention: Improved Corks and Closures

III Applicant:

Name:

Country:

Address:

TAYLOR DETACT APPLICATION FILED 17-02-03  
SECUR 30/09/02 8 340651001

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LONDON WC2B 5SQ 6/77VII Declaration of Priority:  
NilVIII The Application claims an earlier date under Section 5(3), 12(6), 15(4), or 37(4):  
Nil

## IX Check List

A The application contains the following numbers of sheets:

B The application as filed is accompanied by:

- |    |             |          |   |     |
|----|-------------|----------|---|-----|
| 1. | Request     | 1 sheet  | ✓ | nil |
| 2. | Description | 8 sheets | ✓ |     |
| 3. | Claims      | nil      |   |     |
| 4. | Drawings    | nil      |   |     |
| 5. | Abstract    | nil      |   |     |
- 

X It is suggested that Figure No of the drawings should accompany the abstract when published

XI Signature:



Applicant

## Improved Corks and Closures

5 The present invention relates to corks and similar closures.

Corks have traditionally been used to close wine bottles (both still and sparkling), spirit bottles, and the like. The traditional material is natural cork, which is obtained from the bark of certain oak trees. The cork is inserted into the  
10 neck of the bottle and expands due to its inherent elasticity to seal the neck of the bottle, preventing air from ingressing and wine from escaping.

More recently, various modern variants have been introduced. In some cases, these use synthetic plastics materials; in others, they use natural cork which  
15 has been reconstituted in some way, alone or in combination with synthetic materials. Examples of synthetic closures are described, for example, in WO 96/34806. These are typically cylinders of elastomers such as polypropylene, polyethylene and vinyl acetate. The elastomers are melted and mixed with blowing agents which create a foam. The mixture is then typically either injected into a mould or  
20 extruded and sliced to produce synthetic closures of shapes and sizes which are similar to natural corks.

Synthetic closures can also be made of foamed resins such as polystyrene or mixtures of polystyrene and other chemicals such as butadiene rubber. These  
25 are manufactured by injecting beads of expandable polystyrene into moulds. The beads themselves contain a blowing agent, typically pentane. Steam is injected into the mould and the beads swell up and stick together, taking the cylindrical shape of the mould.

30 A comparative trial of synthetic closures versus natural corks, run by the Australian Wine Research Institute and whose results were published in the

Australian Journal of Grape and Wine Research in 2001, showed that in general, synthetic closures do not fully reproduce the sealing capabilities of natural cork. In particular, wine bottled using synthetic closures tends to oxidize faster than wine bottled using quality natural corks. This is partly due to the penetration of oxygen through the interface between the synthetic material and the glass bottle, but also, we have surprisingly found, through the body of the synthetic material. Whilst the rate of oxygen permeation is small, over time the amount of oxygen getting through can be significant.

Our research has found that the polymer chains which make up the elastomers and resins are large molecules which surprisingly provide space for small oxygen molecule to diffuse through. The core of a synthetic closure is foamed to provide much of the elasticity of the closure, so the amount of polymer through which a molecule of oxygen has to pass is small relative to the length of the closure. Different polymers have different rates of permeability to oxygen. The permeability rates for the following polymers commonly found in synthetic closures are as follows:

Polystyrene	933
Polypropylene	1952
Polythene	4669

all in cc/sq m/day for a 1mm thickness. Whilst polystyrene has a better, ie lower, rate of permeability, it tends to suffer from microscopic pores between the fused beads of expandable polystyrene. This allows significantly more gas to pass through the closure than would pass through an extruded or injection moulded closure where no such pores exist.

There have been concerns about the possible permeability of both natural corks and synthetic closures. With natural corks, a wax plug has been used as an additional barrier on the top end of the cork; this is in fact provided primarily for aesthetic effect. With synthetic closures, WO 96/34806 (Betacorque) has pro-

posed the use of a flexible impervious coating such as polyurethane on the ends of the closure if an additional degree of impermeability is required.

The main object of the present invention is to provide a closure member  
5 which provides improved control of gas penetration, particularly oxygen penetration, preferably to a low level.

There are a number of materials which are high oxygen barrier materials. However, in general these materials have properties (such as too low an elasticity,  
10 brittleness, bonding problems, or inadequate as the primary material of synthetic closures) which render them unsatisfactory for present purposes. Many high barrier materials are mainly highly crystalline and hence non-elastic; others are stretchable but with little or no recovery.

15 Some polymers exist which have relatively high gas-barrier capabilities, although they cannot be classified as high gas barriers. Their use for present purposes would therefore preferably require a relatively thick layer. Some of these polymers are based on elastomers such as polypropylene and polyethylene. These incorporate other large molecules within the polymers such as butyl rubber  
20 in an attempt to eliminate the spaces between the polymer chains through which gases can pass. This greatly reduces the passage of oxygen, although it does not eliminate it completely. The incorporation of these additional molecules within the polymers tends to reduce their elasticity, which makes them unsuitable for use as the main constituent of synthetic closures. An example of such a material is  
25 Oxyton CS25, which is produced by DS Chemie. The Mocon oxygen transmission rate for Oxyton CS25 is 500cc through a 100 micron sheet per square metre per day. Such materials are flexible and have a degree of elastic ability but wrinkle when compressed. Similarly, reactive hot melt polyolefines (RHMPs) are relatively high barrier materials but are relatively rigid when set. Other high  
30 barrier materials include vinyl alcohol, ethyl vinyl alcohol (EVOH), polyvinylidene chloride (PVDC), PTFE, polyvinyl chloride, vinylidene chloride-vinyl chloride

copolymer, polyethylene terephthalate, polyamide, oriented polyamide, and modified acrylonitrile. These have barrier properties of between 3 and 6 cc/sq m/day for a 2.5 micron thickness.

5 There are also non-polymeric high barrier materials such as metal foils, vacuum deposited metals, metal oxides and other oxides such as silicon oxide. The latter three are vacuum deposited onto substrates such as polythene to make gas proof bags for food preservation. These have barrier properties of typically 0.5 cc/sq m/day for a 12 micron deposit of aluminium. However, these have  
10 bonding problems and, although they are elastic, have poor resilience.

In summary, no coatings are currently known to exist which provide high levels of gas impermeability combined with elasticity, good adhesion, and the capability of being used in contact with food.

15

According to its main feature the present invention provides a closure member having a low permeability barrier extending across its width, wherein the barrier comprises two layers, the first layer being a low permeability material and the second layer effectively modifying or mediating the properties of the first  
20 layer. (This does not exclude the possibility of there being more than 2 layers.) The second layer may act as an adhesive to improve the adhesion between the first layer and the bulk material of the closure member, and/or modify the mechanical properties of the first layer, as discussed below.

25 The second layer preferably comprises a hot melt (HM) polyolefine adhesive, and more specifically a reactive hot melt polyurethane. Reactive Hot Melt Polyurethanes (RHMPs) are derived from the mixing of two components: an isocyanate rich solution and a polyol-rich solution. (This is in contrast to ordinary polyurethane coatings, which consist of a polyurethane dissolved in a solvent.)  
30 The polyol is mostly polyester and/or acrylic polyols. There are two groups of polyurethanes - aromatic (based on aromatic isocyanates and mostly polyether



polyols) and aliphatic (based on aliphatic isocyanates and /or polyester and acrylic polyols). Aliphatic polyurethanes are preferred, since aromatic polyurethanes have a potential health hazard.

5 With an RHMP, a rapid exothermic chemical polymerisation reaction occurs when the two components are mixed, resulting in a polyurethane pre-polymer. This pre-polymer is what is applied to the closure. It is applied by being melted and spread. The reaction is irreversible under the influence of heat, preventing it from returning to its pre-melted state after use and improving its  
10 barrier properties. Further reactions after application occur under the influence of moisture, resulting in a high level of cross linkage between polymers. The resulting polymer is very stable and has a low level of free monomers which could otherwise taint wine.

15 RHMPs bond very well to polymer based synthetic closures as well as to natural corks. RHMPs are flexible but relatively inelastic when set. However, we have found that when they are stretched or compressed, they become much more elastic. This elasticity allows the gas barrier to move with the closure. In addition, we have found that the close bonding between the layer of RHMP and  
20 the bulk material of the closure results in the elasticity of the bulk material being partially induced or transferred into the RHMP. As a result, the closure can be used in the same way as ordinary corks or synthetic closures, while the RHMP layer forms a relatively low permeability oxygen barrier.

25 Further materials such as metal oxides, eg iron oxide, can be added to the RHMP to improve its oxygen barrier properties.

We have found that there is a further advantage to the use of an RHMP as one layer in the barrier. This elasticity of the RHMP layer provides the force  
30 necessary to achieve the seal required; in addition, where the RHMP layer extends to the edge of the closure, it makes contact with the glass surface and acts as a

gasket preventing gas ingressing and wine exiting along the closure/glass interface. Since it is a substantially denser and tougher material than that of the main synthetic material of the closure, it exerts a considerable extra force on the glass over a small area, so providing a strong seal.

5

In known closures, the barrier is located at an end of the closure member. In the present invention, the barrier may be applied to one or both ends of the closure, but may equally be included in the body of the closure, with the two portions of the closure both being bonded to the barrier to form a unitary closure member.

10

The barrier can be applied anywhere along the length of the body of the closure across the long axis, so that it forms a partial or complete barrier to the movement of gases and fluids. The barrier preferably extends from one side of the closure to the other, thus providing a continuous barrier. However, it can be applied partially, for example only covering the central part and thus providing an improvement to the permeability without providing a complete barrier. It can also be used to provide total coverings. If desired, one or more further barriers may be provided at further positions along the closure.

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The barrier can also extend slightly beyond the cross sectional area, providing a gasket which sits proud of the surface of the closure. Provided that the barrier is elastic enough, the small area of the gasket exerts a high point load around the circumference of the closure/glass interface. This provides a much improved seal to prevent gases passing up the interface. Most synthetics use silicone lubricants, which are not good oxygen barriers, to ensure smooth insertion and extraction characteristics. (In contrast, natural corks usually use paraffin wax or paraffin wax/silicone mixtures, paraffin wax being a good oxygen barrier.)

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The gas barrier layer may be thick or thin. A thin layer is preferable as it takes on the elasticity of the body more effectively and does not look odd on

extraction due to swelling significantly beyond the surface of the closure. However, having a thick gas barrier is more effective at inhibiting gas movement for some oxygen barrier materials.

5 The first layer of the barrier is preferably a material having lower permeability than RHMP. This permits the barrier layer to be thin while achieving very low permeability. Typically the thickness of the RHMP can range from 1 micron up to 2000 microns, with the low permeability material ranging from 1 micron to 1000 microns. Multiple layers of RHMP and barrier materials may be  
10 applied.

The low permeability layer can conveniently be attached to the main body of the closure by the RHMP, and if the barrier is not at the end of the closure, the low permeability layer can conveniently be attached to both parts of the main body  
15 by RHMP layers on both sides.

We have found however that certain types of low permeability material can be formed as a layer directly on the bulk material of the closure. Thus in some cases the barrier can be fixed to the closure by any suitable means such as fusion  
20 by heat, spraying in a suitable solvent, incorporation in the mould, physical bonding, chemical bonding, vapour deposition, or melting and application by printing or rolling on. A barrier which is applied as a liquid can be applied by any means such as direct application, brushing, printing, spraying, or dipping. In most such cases, however, an RHMP layer can also be used. If the barrier is not at the end of  
25 the closure, the RHMP secures the open side of the low permeability layer to the second half of the closure; if the barrier is at the end of the closure, the RHMP layer is applied to its outer surface.

Where there is an RHMP layer between the low permeability layer and the  
30 substrate, it ensures good adhesion between the low permeability layer and the substrate. In addition to this direct effect, the RHMP layer aids in effectively

transferring or inducing the elastic properties of the substrate into the low permeability layer. The more powerful elastic response of the RHMP, combined with its strong adhesive capabilities, forces the low permeability layer to conform with the movements of the RHMP layer. The chance of fracture in a low permeability layer of brittle material is thus greatly reduced. (Further, if such fracture should occur, its extent is likely to be greatly reduced, and its effects minimized as the RHMP will keep the pieces in place.)

Combinations of different low permeability materials may be used to achieve high barrier capabilities for varying requirements. For example, a metal layer can be deposited onto the end of the closure and then covered by a co-extruded EVOH film.

One or both layers may be in the form of a free-standing film which is applied to the closure. In particular, it is possible to use a single such film having a plurality of layers, one of which is a low permeability material and another of which can be used as a HM layer. Such films are currently used in "free-standing" applications such as for food packaging. (They are generally made by co-extrusion of the different layers together with the application of adhesives between the layers where that is necessary to bond the layers together.) A typical such film, for example, consists of 3 layers of polyethylene, UVOH, and nylon; this can be applied for present purposes with the polyethylene layer acting as a hot melt material.

The present invention may be contrasted with the closures of WO 96/34806 patent mentioned above. That described only polyurethane resins as sealants, not 2-layer barriers, nor the use of hot melt (HM) polyolefines or reactive hot melt polyurethanes (RHMPs). At that time, the movement of oxygen through materials was not understood and the resins were designed only to stop the gross movement of gas and moisture.

The present system can be applied to natural corks as well as synthetic closures to improve their gas permeability. In the case of natural corks, the present barrier will also act as a barrier to microbiological contaminants such as cork-taint (eg trichloroanisole (TCA)) and yeasts which contaminate wine, as well as acting  
5 as a barrier to chemicals in the cork which could otherwise enter the wine such as tannins and tars.

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### Claims

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Any feature of novelty or combination thereof within the meaning of Article 4H of the International Convention (Paris Convention).

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